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Charles University
Institute of Physics
PRAGUE 2 CZ-121 16
Czech Republic

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14. ABSTRACT

This report results from a contract tasking Charles University as follows: The contractor will investigate the production of CdTe/CdZnTe crystals for use as substrates for epitaxial growth of HgCdTe layers for infrared focal plane array detectors. The contractor will develop a process for producing surfaces on CdZnTe wafers with surface finish (Ra) of 7 nm or less, flatness of l micron or less, and parallelism of 1 micron or less, using a wafer polishing machine.

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Final report

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Issuing organization and point of contact

Prof.Pavel Höschl

Institute of Physics, Charles University

Ke Karlovu 5

CZ-121 16

Czech Republic

Fax (+420 2 24922797)

Distribution:

Lt.Col.C.A.Kutsche	EOARD	1 copy
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Content:

1. Introduction

The final report describes the results of research and development of CdZnTe crystals for use as substrates for epitaxial growth of HgCdTe for infrared focal plane arrays. The effort built up on the results received during the foregoing projects and concentrated mainly on introducing a new setup for mechanical-chemical polishing and development of polishing process resulting in surface quality suitable for MBE growth of epitaxial layers. Results of a comparative study of surface quality of samples from commercial vendor (Nikko, Japan) and from our laboratory by several methods are reported. Three samples of substrates fabricated from CZT crystals grown in our laboratory by vertical gradient freeze method (VGF) were delivered to NVESD (Fort Belvoir, VA). The final polishing was performed using the new chemical-mechanical polishing setup (Logitech, Scotland) purchased within the framework of this project.

The efficiency of using ultra high purity quartz and 7N starting elements in reducing the level of impurities in CdZnTe crystals was investigated and the results are documented by Glow Discharge Mass Spectroscopy (GDMS) and photoluminescence measurements.

The contact with prof. Sivalingham Sivanathan from University of Chicago, IL was established. Prof. Sivanathan and Dr.Paul Boieriu from EPIR Ltd visited the Institute of Physics, Charles University in Prague in March 2003. The results of the current status of activities on CZT substrate fabrication and polishing were discussed during the visit. Experiments aimed at repolishing of used substrates from commercial vendors using our mechanical and chemical polishing procedure were agreed. These experiments were performed during the second visit of Dr.Paul Boieriu and co-workers in June 2003 in Prague.

2. Development of surface polishing treatment

Development of the surface polishing treatment was started after putting the new Chemical Polishing Machine CP3000 (Logitech, Scotland). It includes robust testing of the surface quality after each polishing step. We used the inteferometer Zygo (USA), model New View 5020, which proved to be very suitable for testing of technological operations during the polishing. This interferometer is located in a development laboratory of company Preciosa located approx 150km from Prague. This setup enables to scan large areas of substrates up to

3.5x2.6mm^2 during one measurement. The smallest scanned area is 70x50μm^2. In addition, Atomic Force Microscope (AFM) was used to test the surface quality on small areas.

The final goal of long-term polishing effort is to reach surface roughness comparable or better than the commercially available substrates (Acrotec-Nikko, Japan). Therefore we performed a comparative study of several substrates purchased from Nikko in order to characterize the standard Nikko quality. The results of measurements of surface roughness are given in Table 1. Examples of measurements on the optical interferometer are shown in Figs 1-11.

Several conclusions can be done. The parameters characterizing surface roughness (rms, Ra) depend on the scanned area or length of scanned line. The higher the area or the longer the line, the higher the value of rms or Ra. Therefore comparison of different substrates can be done only using the same area or line length. The rms parameter of three Japanese substrates (Nikko) measured on the large area 3.57x2.68mm^2 is in the range of 5-14 nm. The substrate from our laboratory polished by the developed polishing technology has the rms value 8-6nm, which is only slightly higher than typical value of Nikko substrates. Detailed examination of Figs. 1-4 shows, that all studied Nikko substrates exhibit similar surface morphology characterized by a relief slowly varying at the distance of 1-2 mm. This relief substantially contributes to the measured rms value causing its relatively high values in some cases (14nm). This value is certainly dependent also on the success of applied numerical corrections. Therefore we conclude, that the value of rms=5 nm is typical for Nikko substrates measured at area 3.57x2.68mm^2 The 122B61, F13F84 and 122B43 Czech substrates do not show such a relief, which indicates their better flatness. It means, however, that the rms value of 6nm is caused mainly by shorter order variations with a period of ~hundreds of μm (see linescan in Fig.6). Linescan in Figs.7 and 9 demonstrate, that at the distance of tens of μm the rms value is well below one nm. It seems reasonable to assume, that this short range roughnes should be the most important one from the point of view of quality of the MBE layer.

We conclude, that the quality of polishing developed in our laboratory is approaching the original quality of Nikko substrates. At short range distance the rms value well below one 1nm was reached. Certain differences in the surface morphology as a result of the Nikko polishing procedure and our one are present.

Sample	Source	Area/line	Rms(nm)	Ra(nm)
AZ813	Nikko	3.57x2.68mm^2	14	11
		2.5mm	6	5
		1mm	1	1
SU910 Place 1	Nikko	3.57x2.68mm^2	14	11
		2.5 mm	4	4
SU910 Place 2	Nikko	3.57x2.68mm^2	5	4
		2.5 mm	2	1
		0.7mm	1	1
SZ819	Nikko	3.57x2.68mm^2	5	4
		70x50μm^2	<1	<1
		2.5mm	2	2
		700 μm	<1	0.37
		40 μm	<1	0.18
122B61	Charles University	3.57x2.68mm^2	8	6
		70x50μm^2	1	1
		2.5mm	8	6
		30 μm	<1	0.16
F13F84	Charles University	3.57x2.68mm^2	11	9
		70x50μm^2	1	1
		2.5mm	8	6
		30 μm	<1	0.19
122B43	Charles University	3.57x2.68mm^2	8	6
		70x50μm^2	2	1
		2.5mm	7	5
		30 μm	<1	0.16

Table 1 Comparison of original surface quality of commercial substrates and a Charles University substrates polished by the developed polishing technology

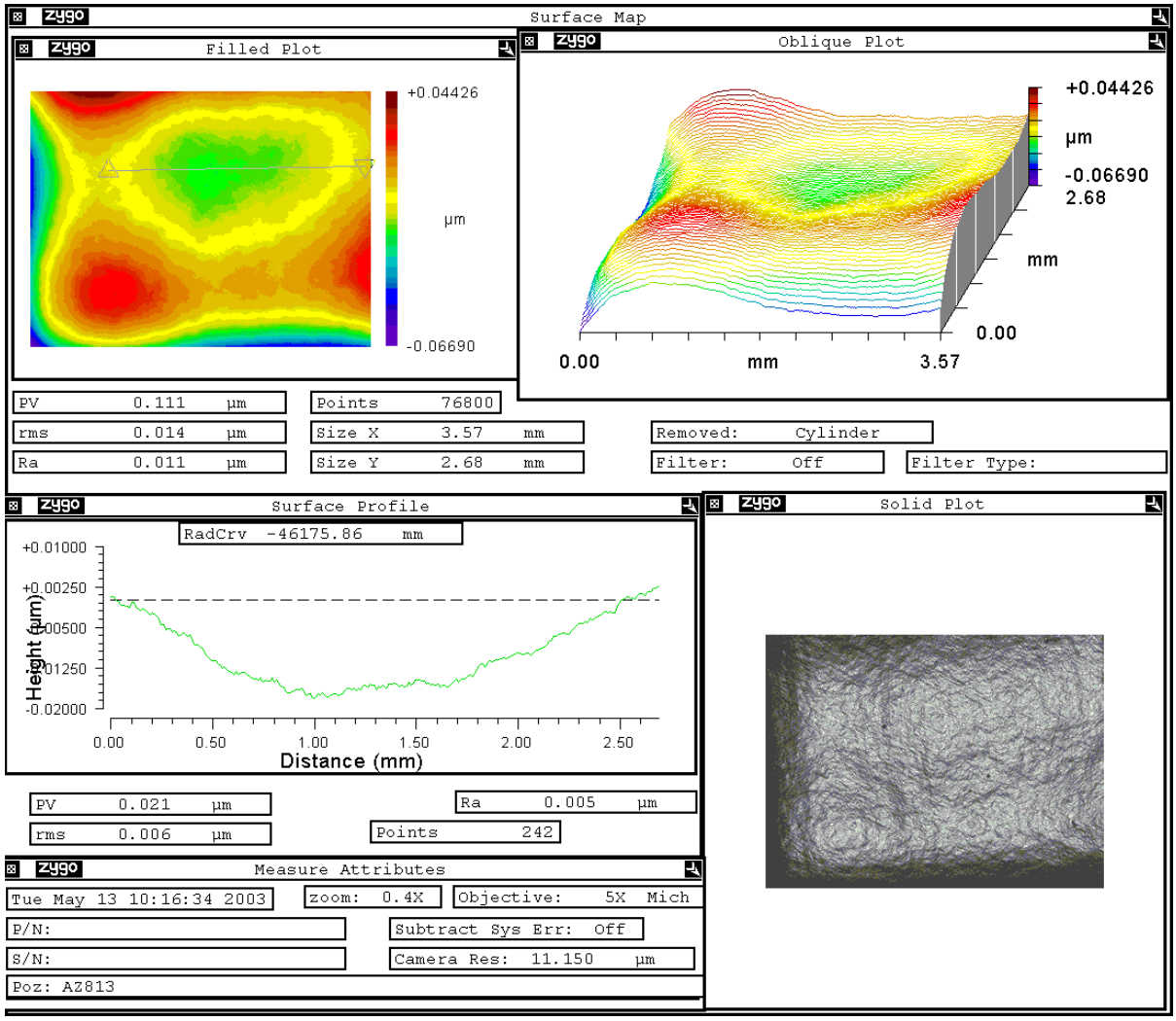

Fig.1 Surface profiles of sample AZ813 (Nikko)

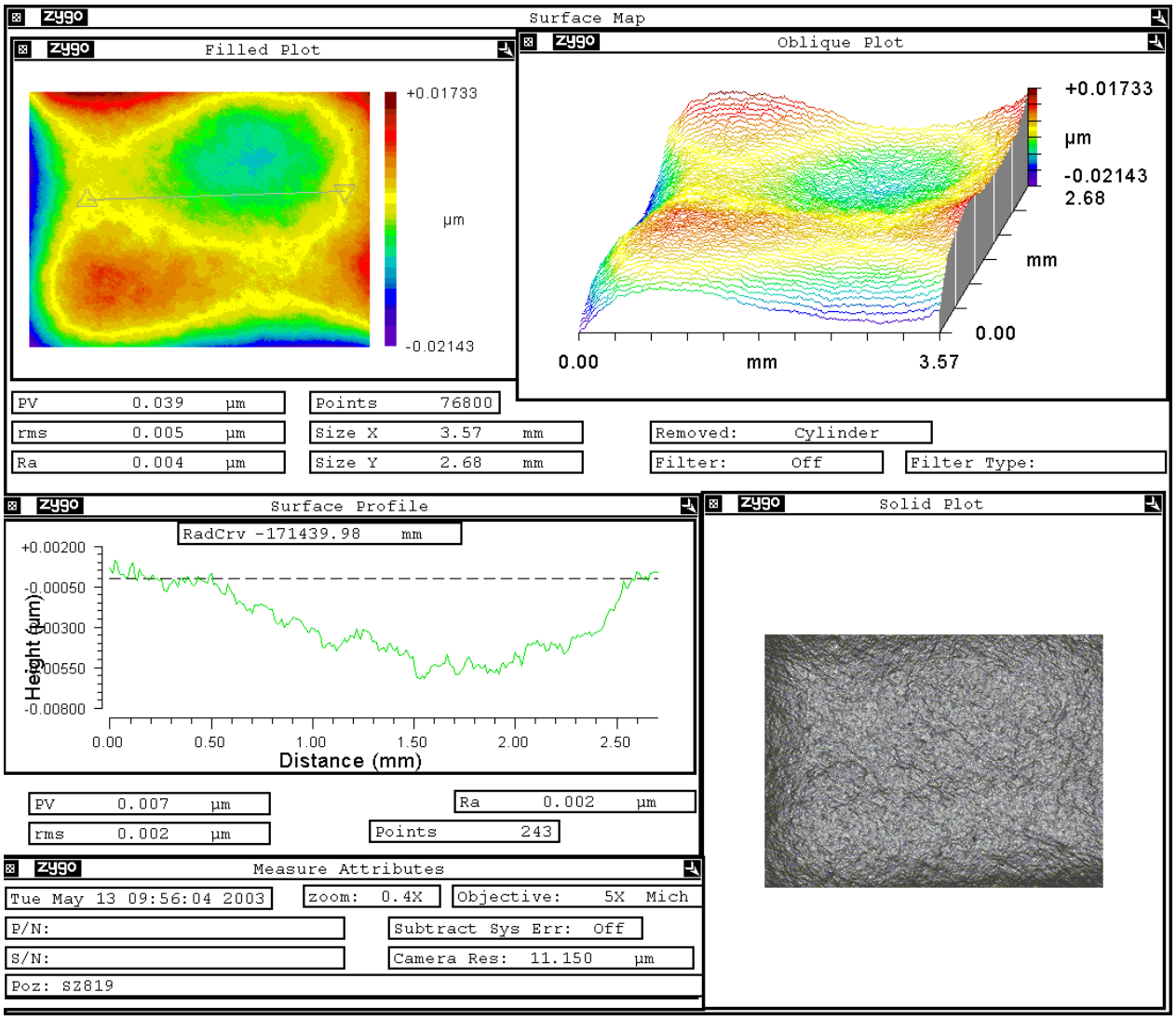

Fig2 Surface profiles of sample SU910 (Nikko) Place 1

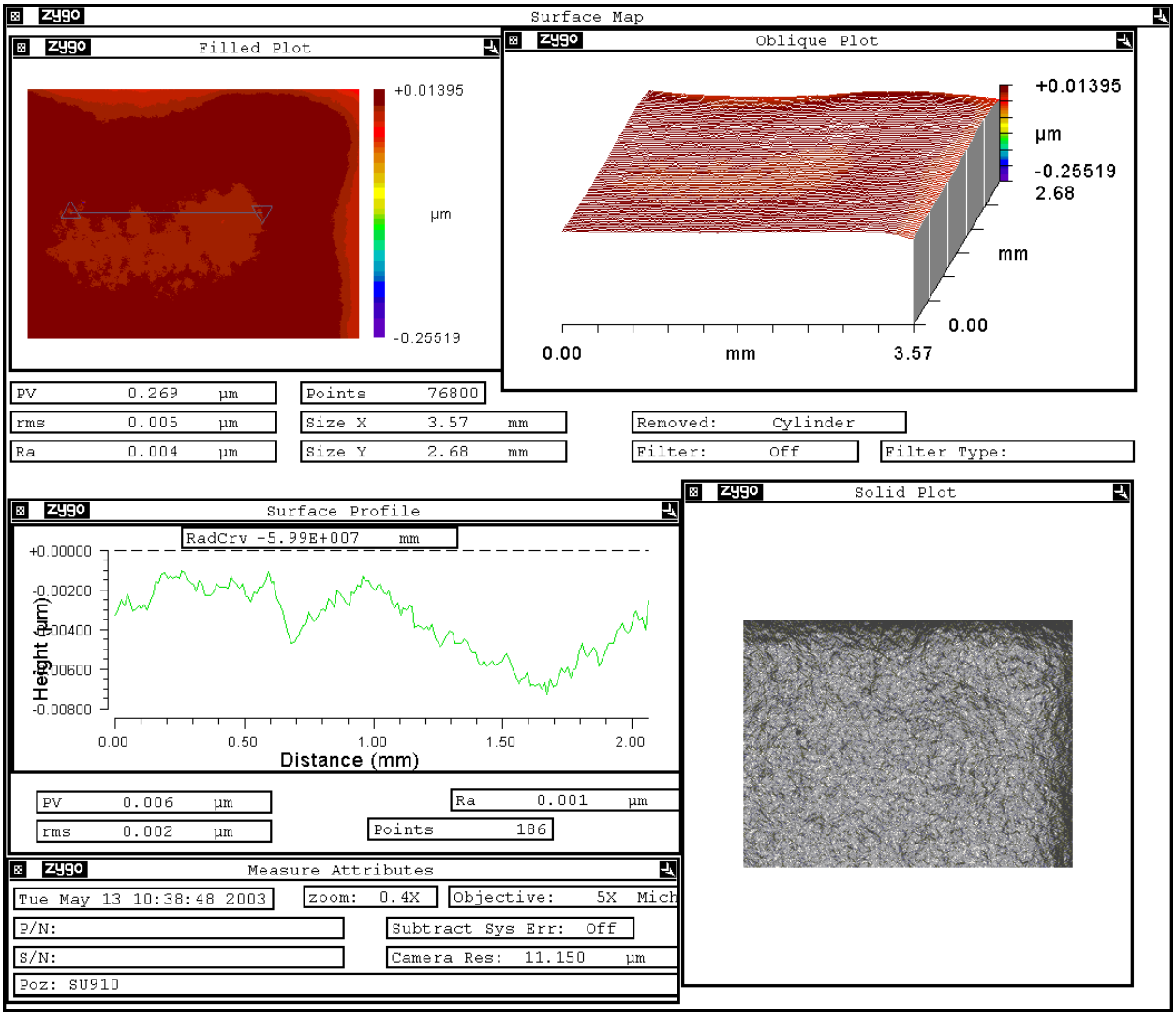

Fig3 Surface profiles of sample SU910 (Nikko) Place 2

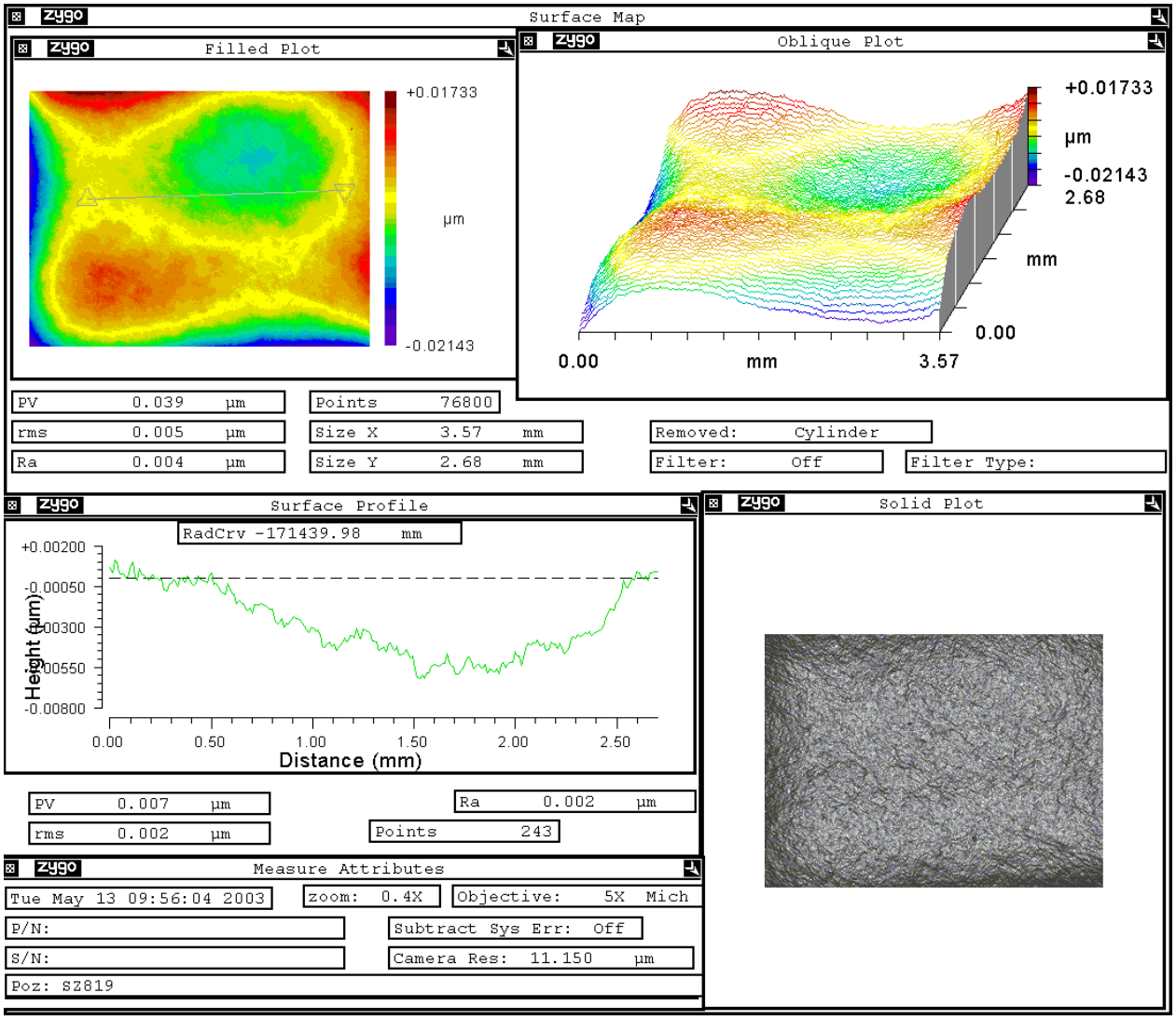

Fig.4 Surface profile of sample SZ819 (Nikko)

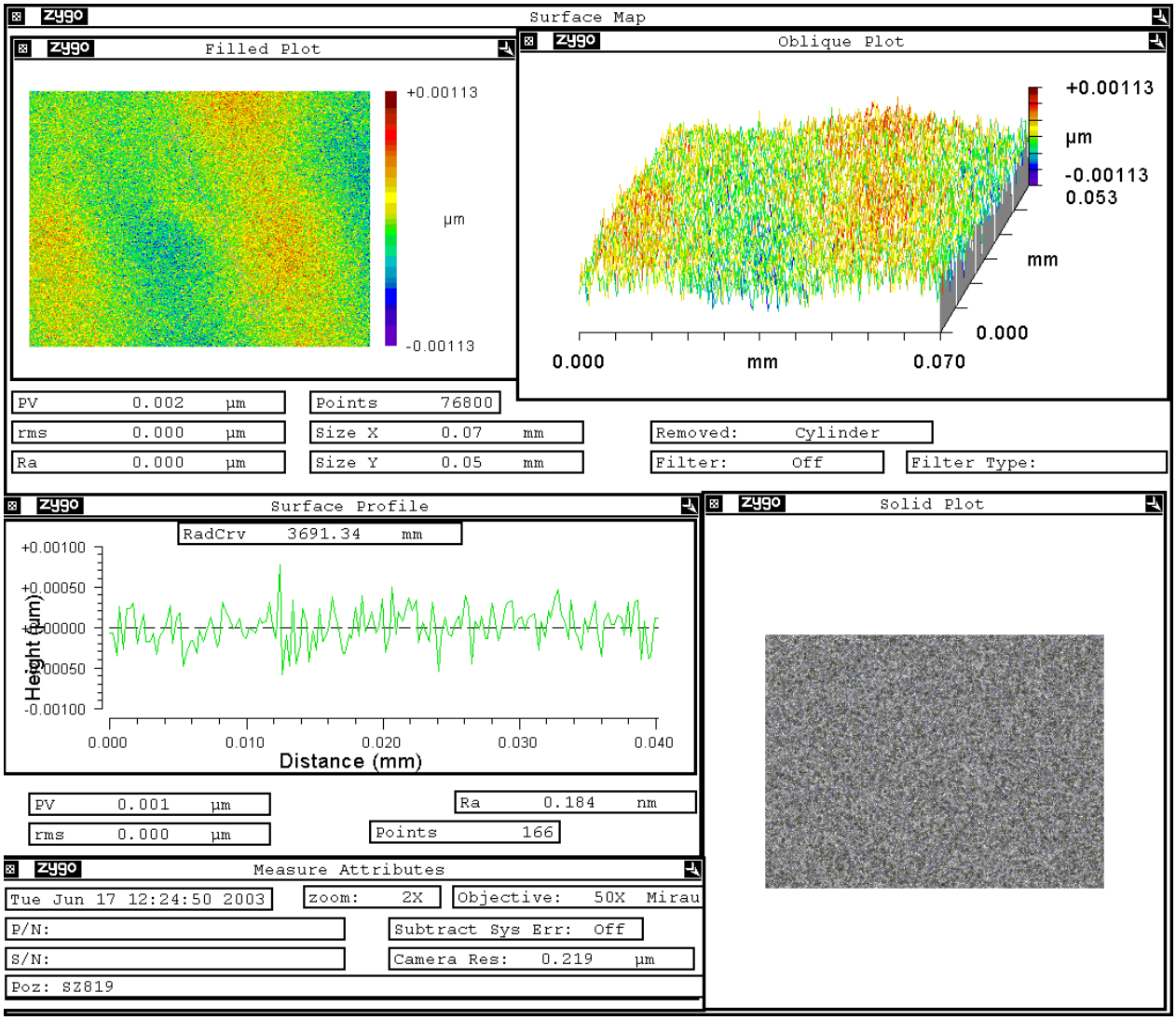

Fig.5 Surface profile of sample SZ819 (Nikko) - detail

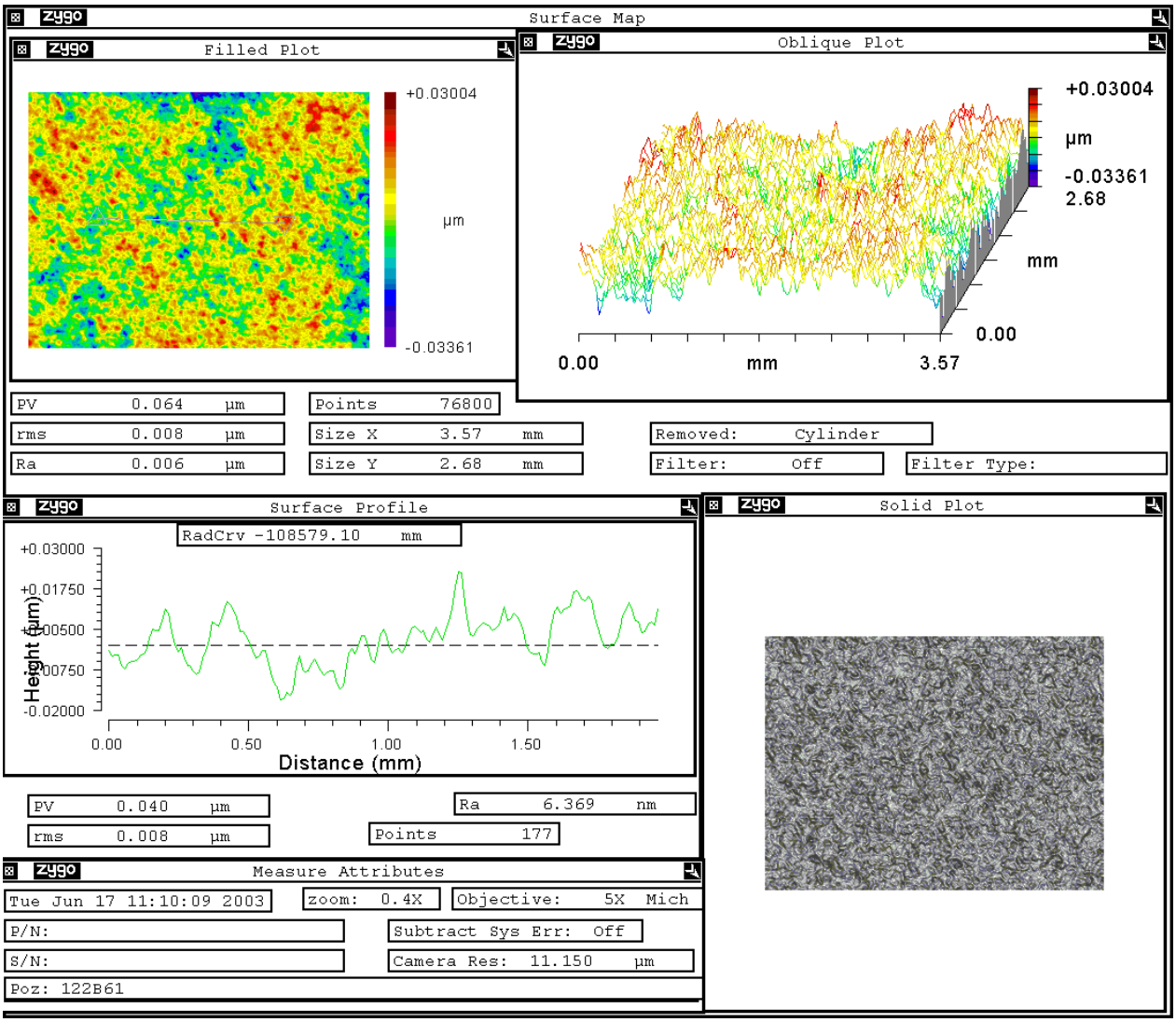

Fig.6 Surface profile of sample 122b61 (Charles University)

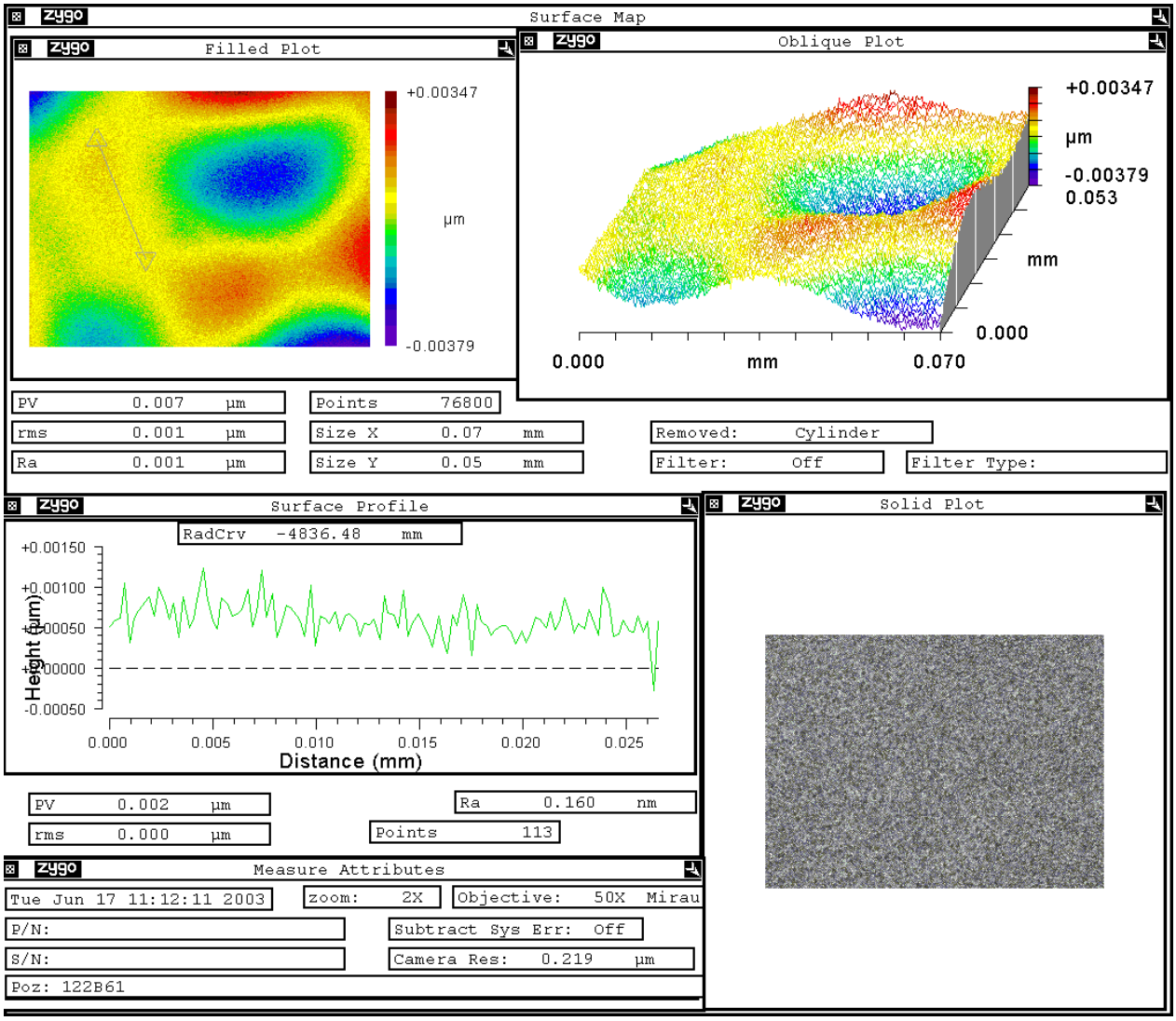

Fig.7 Surface profiles of sample B61(Charles University)-smaller area

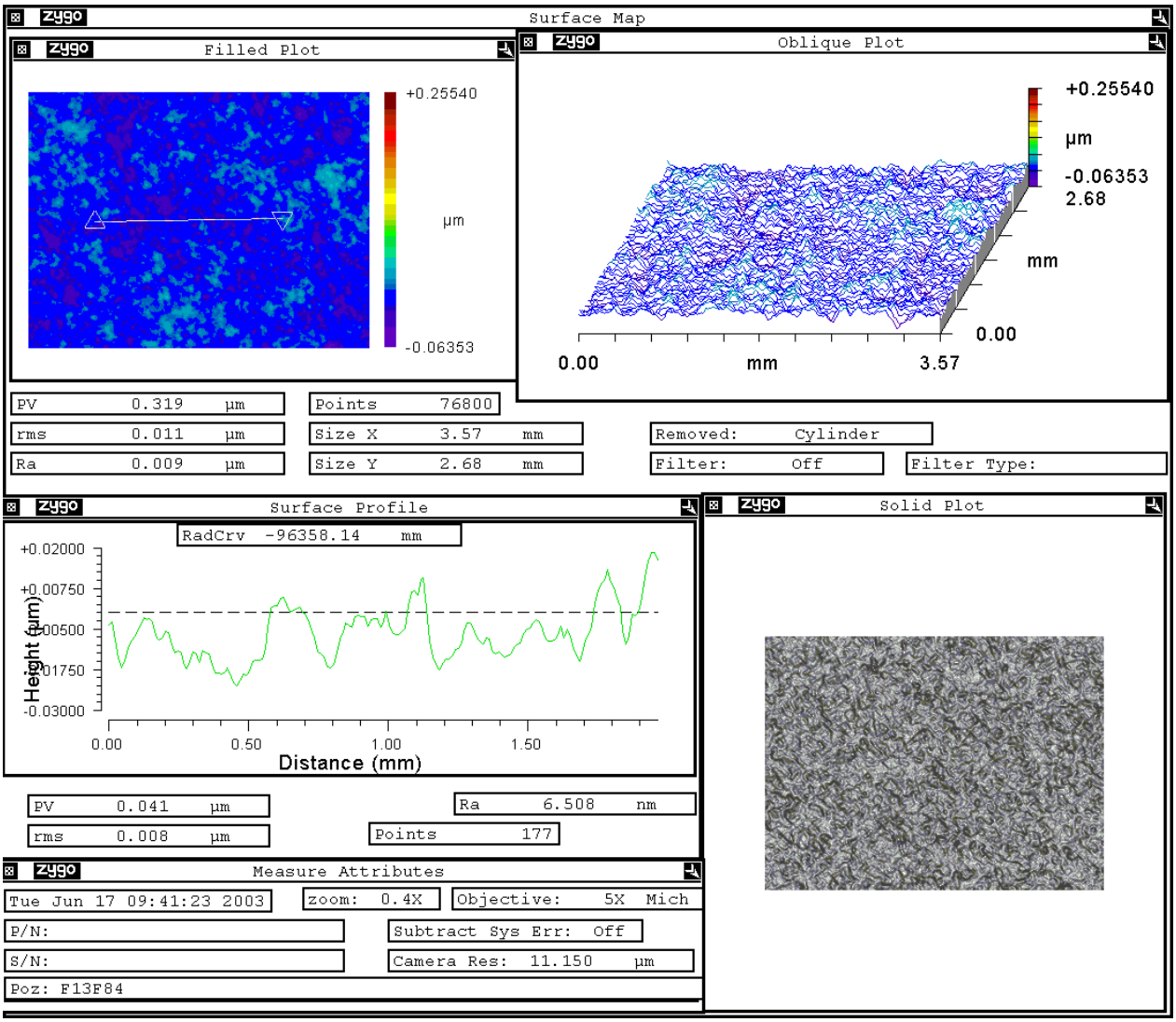

Fig.8 Surface profile of sample F13F84 (Charles University) - detail

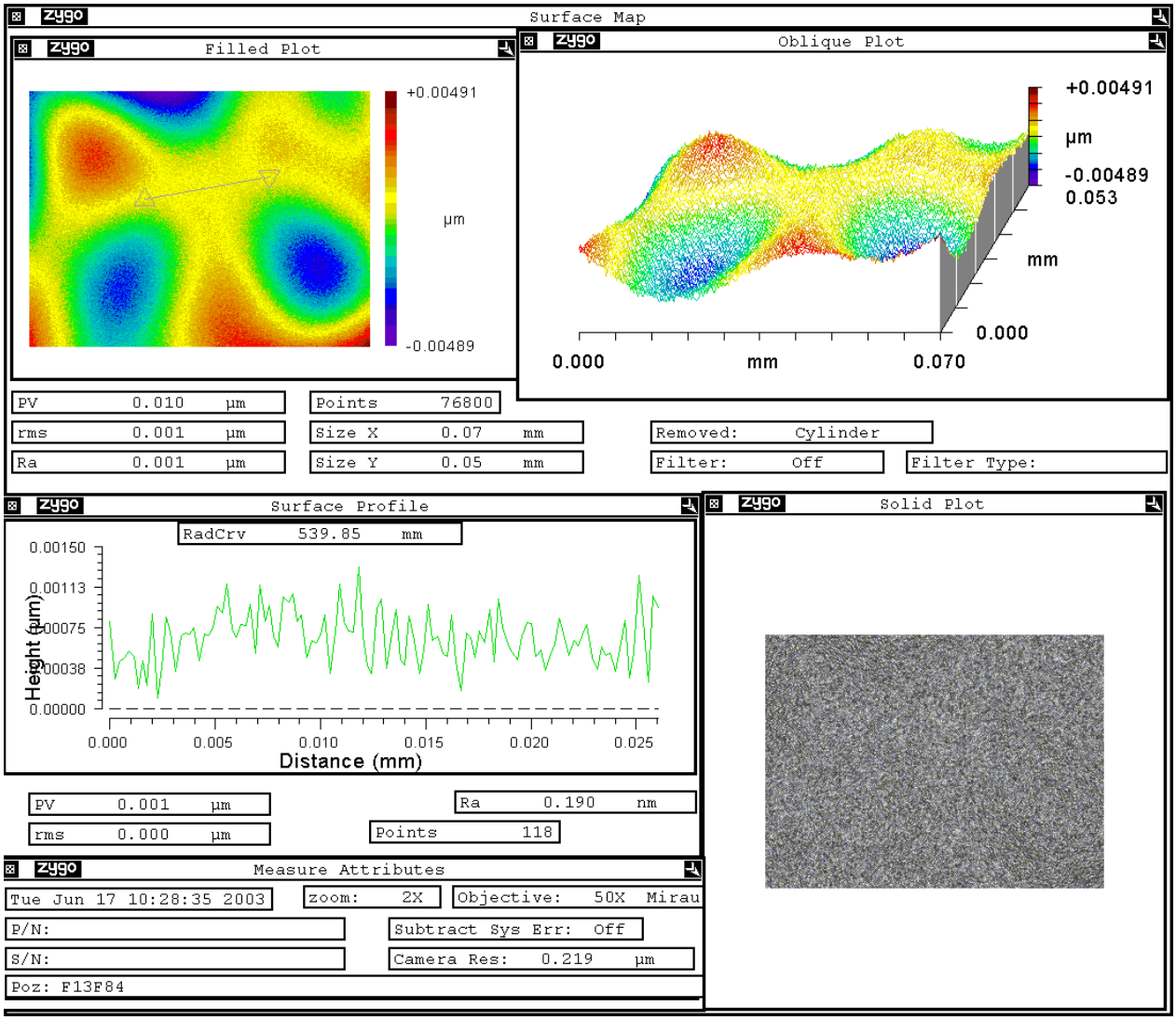

Fig.9 Surface profile of sample f13f84 (Charles University) – detail

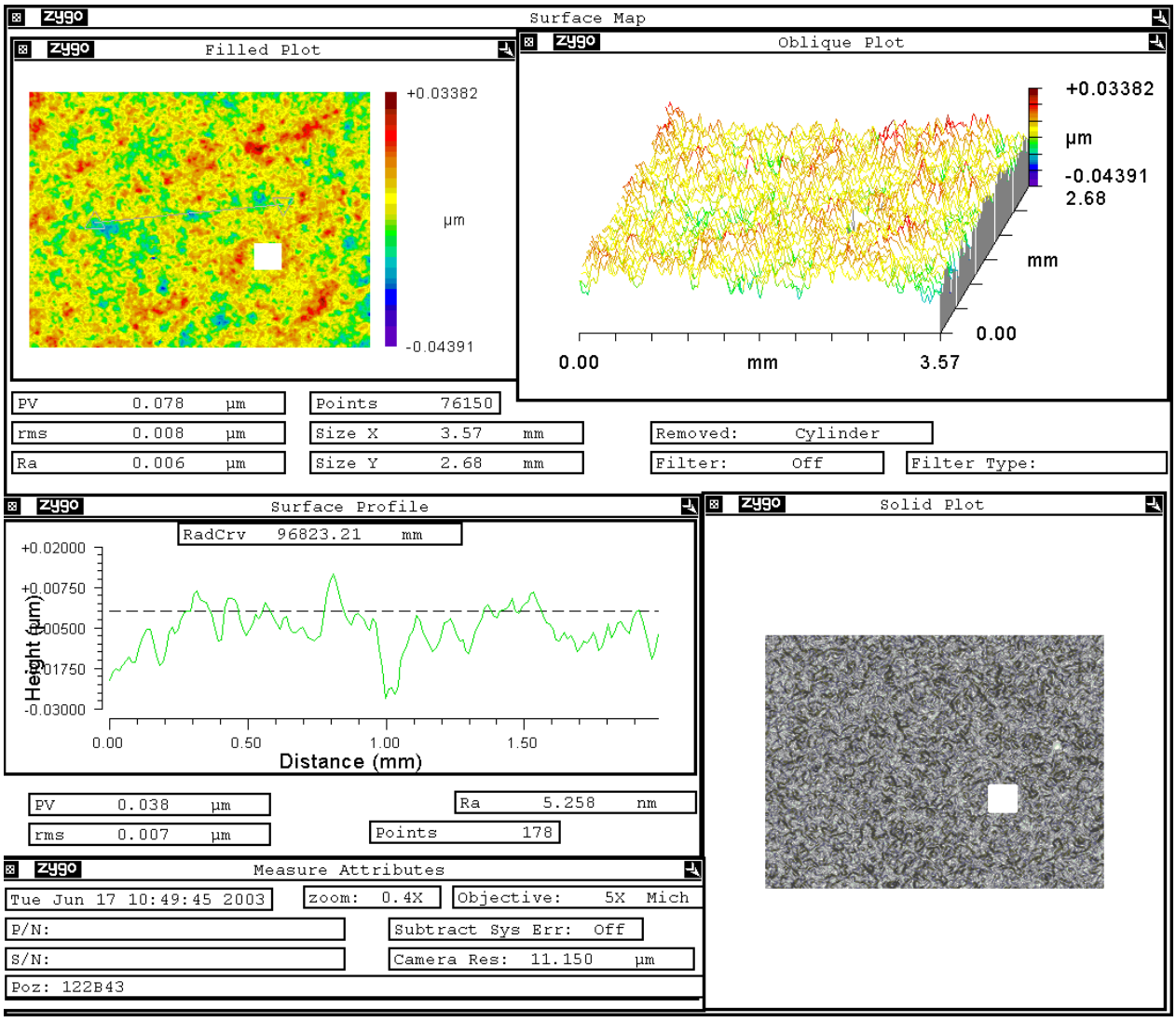

Fig.10 Surface profile of sample 122B43 (Charles University)

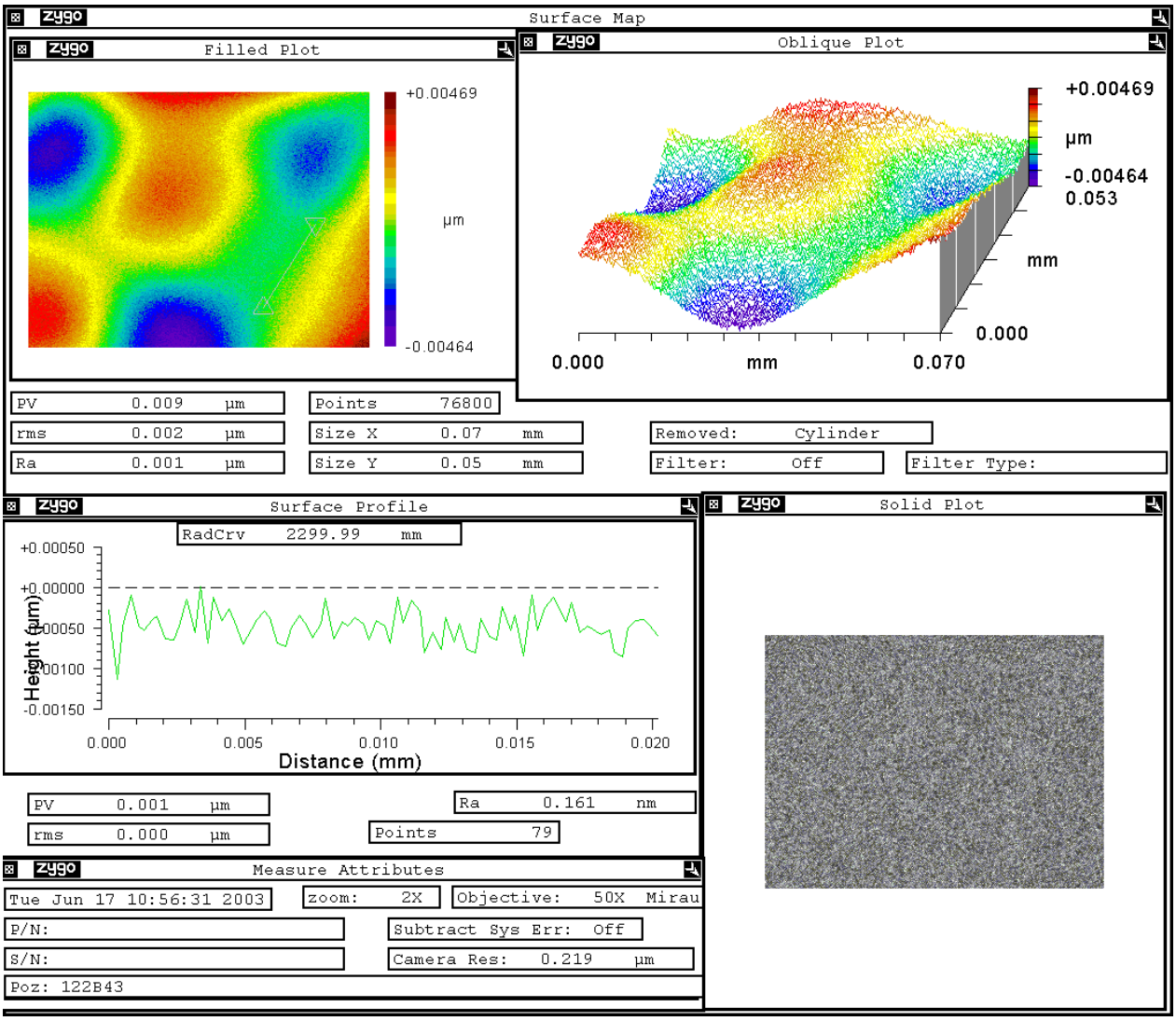

Fig.11 Surface profile of sample 122b43 (Charles University) – detail

3. Substrate fabrication

The substrates were cutted and oriented by the procedure described in the Final report of the project F61775-O1-WE004. The polishing was performed by a developed procedure described in the previos section. Three substrates are delivered to NVESD. Results of rocking curve measurements on two of the delivered samples are presented on Figs. 14-15. The scanned area was 6x2mm^2.

4. Investigation of influence of quartz and starting elements purity on impurity

An increased content of some foreign impurities (mainly alcali metals and Fe) was found in the past in crystals prepared in our laboratory. This question was more deeply discussed in the Final report of project F61775-O1-WE004. Therefore we performed growth of a crystal from 7N starting elements and using quartz of higher purity. The technology of quartz handling was modified in order to exclude contamination during sealing off the ampoules. For this study a smaller crystal of diameter 20mm was used. The results of GDMS analysis of one sample from this crystal in comparison with three crystals prepared from 6N starting elements and lower purity quartz are shown in Table 1. The influence of the purification of the whole process is eveident. The content of the critical elements (Na, Mg, B) decreased by 1-2 orders of magnitude to the level of several ppb (1ppb=3.10^{13} cm^{-3}). Also the content of Cu and Fe decreased to a very low level 20-30ppb.

The purification effect can be demonstrated also by low temperature photoluminescence measurements. Fig.14 shows a comparative PL measurement of our sample fabricated from 6N starting elements and two commercial ones (Acrotec-Nikko), where the higher intensity of donor-acceptor pair recombination (DAP) of our sample in comparison to commercial ones indicated higher content of shallow impurities in our samples. Fig.15 shows PL spectrum (4.2K) of crystal D (Table 1). Apparently, the spectrum is dominated by peaks of bound excitons, the PL intensity of the defect band (1.4eV) being very low. This is in good correlation with the GDMS results presented in Table 2.

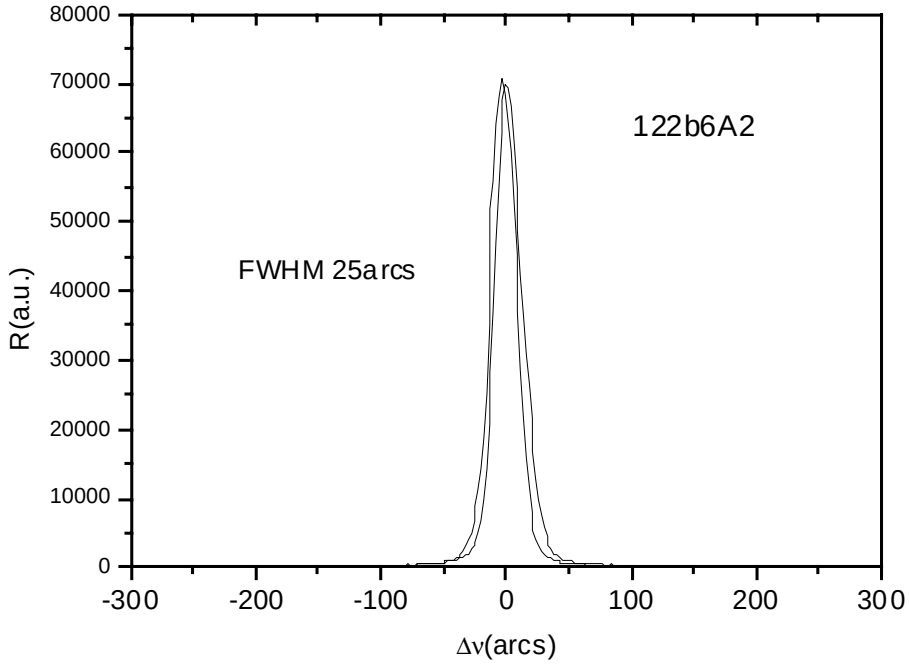

Fig.12 Rocking curves of sample 122b61 measured at two positions (distance 4mm). Scanned area 6x2 mm^2

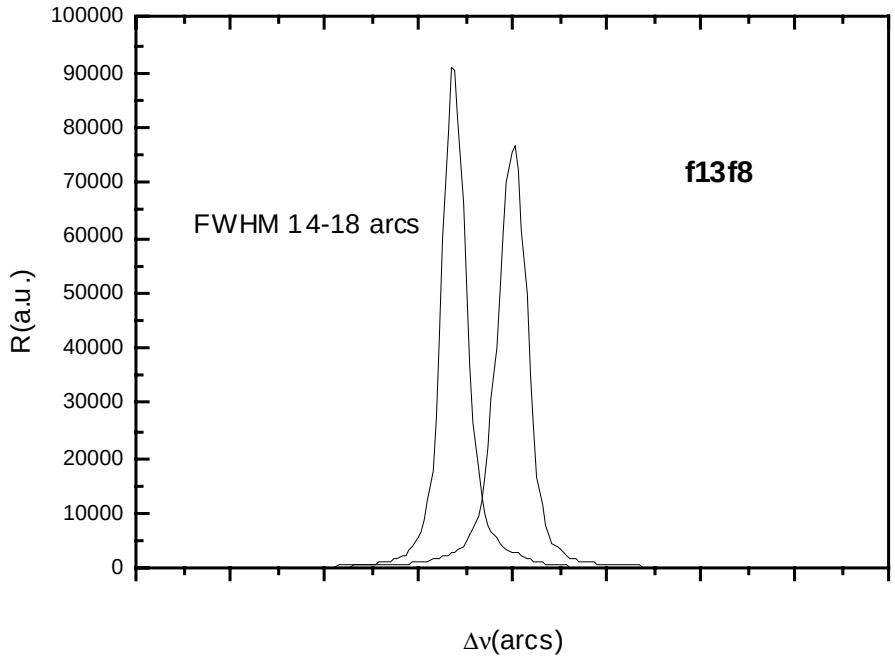

Fig.13 Rocking curves of sample F13F84 measured at two positions (distance 4mm). Scanned area 6x2 mm^2

Table 2

GDMS analysis of crystals grown from 6N and 7N elements and different quartz quality

N	Sample Type	A 6N	B 6N	C 6N	D 7N
	Element	Standard quartz	Standard quartz	Standard quartz	High purity quartz
1	Li	< 2	< 1.5	< 1.6	< 1.8
2	B	20	890	220	7
3	Na	10	130	100	2
4	Mg	120	25	50	8
5	Al	150	5	8	< 0.5
6	Si	8	8	43	< 4
7	P	20	< 0.4	12	1
8	S	110	48	48	9
9	K	< 7	< 7	< 3	< 5
10	Ca	< 20	< 12	< 15	< 15
11	V	< 0.5	< 0.1	< 0.1	< 0.2
12	Cr	23	23	5	7
13	Mn	< 15	< 16	< 10	< 17
14	Fe	300	100	26	30
15	Co	< 0.2	1.7	< 0.2	< 0.3
16	Ni	120	40	0.7	1.7
17	Cu	< 20	180	30	20
18	Zn	4.5%	10	2	20
19	Ge	50	< 8	< 6	<8
20	As	< 50	< 100	< 70	<100
21	Se	< 15	< 6	< 6	<7
22	Br	< 7	< 7	< 8	<12
23	Ag	< 80	< 15	< 10	<13
24	In	< 25	< 20	< 15	0
25	Sn	< 25	<20	<30	<30
26	Sb	< 20	< 16	< 13	<25
27	Hg	< 2	< 1	< 0.7	<3
28	Tl	< 0.2	< 0.2	< 0.1	<0.2
29	Pb	< 0.4	< 0.3	0.4	<1
30	Bi	< 0.1	< 0.1	< 0.1	<0.3

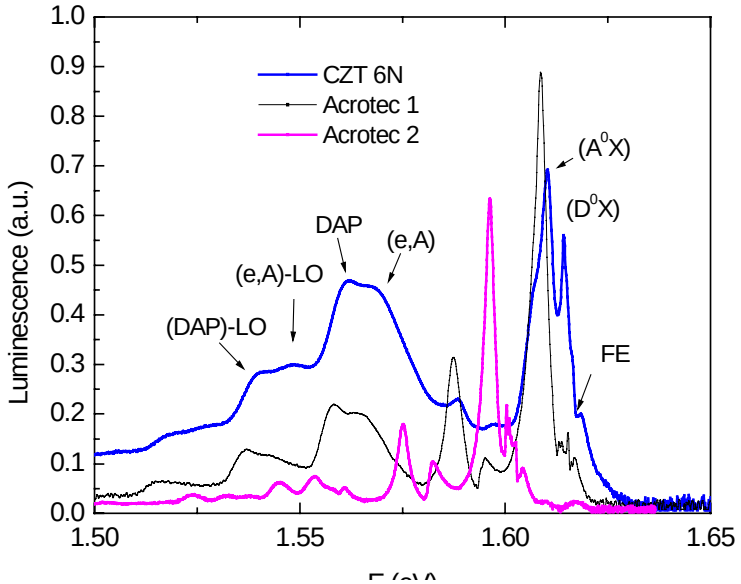

Fig 14.Photoluminescence of two CZT Acrotec (Nikko) sample and one CZT Prague sample prepared from 6N materials

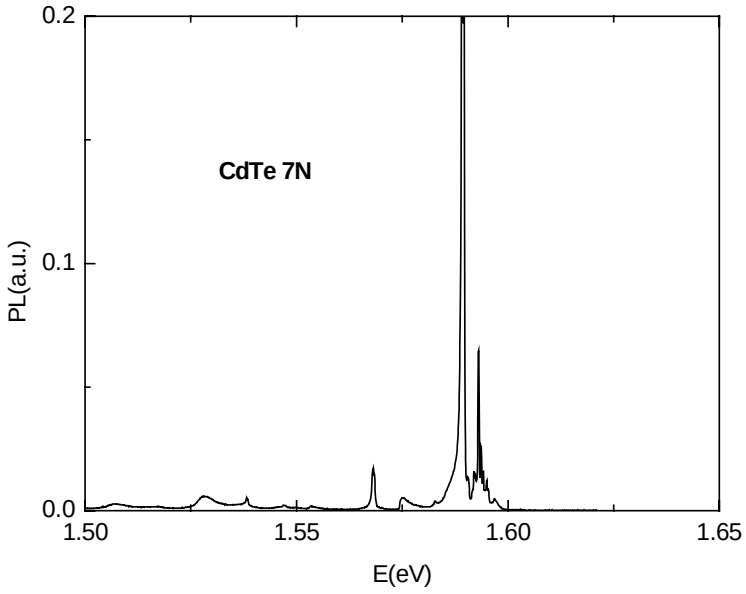

Fig.15 Photoluminescence of CdTe sample fabricated from 7N materials